PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Norimitsu KAIMAI, et al.

Docket No: Q95282

Appln. No.: 10/596,171

Group Art Unit: 1795

Confirmation No.: 3140

Examiner: Jane J. RHEE

Filed: February 8, 2007

For: MICROPOROUS COMPOSITE MEMBRANE AND ITS PRODUCING METHOD AND USE

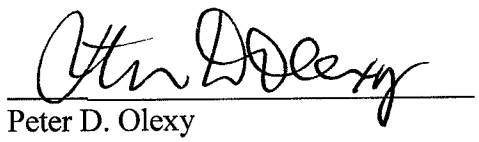
COMPARATIVE EXPERIMENTS

Commissioner:

Attached herein is a three-sheet document received from Applicants wherein one of the Inventors of the present application has performed certain experimentation.

That experimentation is discussed in the AMENDMENT...1.111 submitted herewith.

Respectfully submitted,



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COMPARATIVE EXPERIMENTS

I am one of the inventors and conducted the following experiments.

To compare the microporous composite membrane meeting the requirements of the present invention with those produced by using polypropylene failing to meet the requirements of the present invention, the following experiments were conducted.

Experiment 1

To prepare a polypropylene having solubility of less than 0.5 g in 100 g of toluene at 25°C but meeting the requirement of the racemic diad fraction, 3 g of polypropylene used in Example 1 and 27 g of polypropylene used in Comparative Example 5 were mixed. The resultant polypropylene composition had Mw of 73,000, Mw/Mn of 1.8, solubility of less than 0.1 g in 100 g of toluene at 25°C, and a racemic diad fraction [r] of 0.80. Toluene was added to the polypropylene composition to a concentration of 0.5% by mass, and stirring was conducted at room temperature for 12 hours. However, the polypropylene composition was not dissolved in toluene.

Experiment 2

To prepare polypropylene having a racemic diad fraction [r] of less than 0.16 but meeting the requirement of the solubility, 15 g of polypropylene prepared in the same manner as in Comparative Example 6 except for controlling the Mw to 20,000 by changing the amount of a hydrogen gas to 2,000 mL was mixed with 15 g of the polypropylene used in Example 1. The resultant polypropylene composition had Mw of 30,000, Mw/Mn of 3.0, solubility of 10 g or more in 100 g of toluene at 25°C, and a racemic diad fraction [r] of 0.14. Toluene was added to the polypropylene composition to a concentration of 9.1% by mass, and stirring

was conducted at room temperature for 12 hours. Using the resultant solution of the polypropylene composition in toluene as a coating liquid, a microporous composite membrane was produced in the same manner as in Example 1. Observation with the naked eye revealed that the coating layer had poor uniformity. The properties of the microporous composite membrane are shown in Table A below.

Experiment 3

To prepare polypropylene having a racemic diad fraction [r] of more than 0.84 but meeting the requirement of the solubility, 15 g of the polypropylene used in Example 5 and 15 g of the polypropylene used in Comparative Example 5 were mixed. The resultant polypropylene composition had Mw of 120,000, Mw/Mn of 2.2, solubility of 13 g or more in 100 g of toluene at 25°C, and a racemic diad fraction [r] of 0.88. Toluene was added to the polypropylene composition to a concentration of 11.5% by mass, and stirring was conducted at room temperature for 12 hours. Using the resultant solution of the polypropylene composition in toluene as a coating liquid, a microporous composite membrane was produced in the same manner as in Example 1. Observation with the naked eye revealed that the coating layer had poor uniformity. The properties of the microporous composite membrane are shown in Table A below.

Table A

Properties	Requirements	Experiment 1	Experiment 2	Experiment 3
Coating Layer				
Racemic Diad Fraction [r] of Polypropylene	0.16-0.84	0.80	0.14	0.88
Solubility (g) of Polypropylene in 100 g of Toluene at 25°C	≥ 0.5	< 0.1	≥ 10	13
Grams per 1 m^2 of Microporous Polyethylene Membrane	0.1-5	-	2.0	2.0
Microporous Composite Membrane				
Porosity (%)	25-95	-	25.5	23.5
Air Permeability (seconds/100 cc)	100-3,000	-	11,500	12,300
Shutdown Temperature (°C)	120-140	-	135	135
Meltdown Temperature (°C)	≥ 155	-	160	160
Battery Capacity Recovery Ratio (%)	≥ 70	-	60	55

Polypropylene having solubility of less than 0.5 g in 100 g of toluene at a temperature of 25°C did not form a coating layer (Experiment 1). Polypropylene having a racemic diad fraction less than 0.16 or more than 0.84 failed to provide a microporous composite membrane having a uniform coating layer and a battery capacity recovery ratio of 70% or more (Experiments 2 and 3). Accordingly, it is concluded that microporous composite membranes meeting the requirements of the present invention can be obtained only by using polypropylene having racemic diad fraction [r] within a range of 0.16-0.84, and solubility of 0.5 g or more in 100 g of toluene at a temperature of 25°C.

Dated: March 16, 2009

KAIMAI Norimitsu